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Plastics — Determination of fracture toughness (G_{IC} and K_{IC}) at moderately high loading rates (1 m/s)

Plastiques — Détermination de la ténacité à la rupture (G_{IC} et K_{IC}) à vitesses de charge modérément élevées (1 m/s)

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical behaviour*.

This second edition cancels and replaces the first edition (ISO 17281:2002), which has been technically revised with the addition of Annex C.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document is based on a testing protocol developed by the European Structural Integrity Society (ESIS), Technical Committee 4, *Polymers, Polymer Composites and Adhesives*, who carried out the preliminary enabling research through a series of round-robin exercises which covered a range of material samples, specimen geometries, test instruments and operational conditions (see References [3] to [6]). This activity involved about 30 laboratories from 12 countries.

Plastics — Determination of fracture toughness (G_{IC} and K_{IC}) at moderately high loading rates (1 m/s)

1 Scope

This document specifies the principles and provides guidelines for determining the fracture toughness of plastics in the crack-opening mode (Mode I) by a linear elastic fracture mechanics (LEMF) approach, at load-point displacement rates of up to 1 m/s. It supplements ISO 13586 so as to extend its applicability to loading rates somewhat higher than is the case in the scope of the latter document.

Fracture testing at high loading rates presents special problems because of the presence of dynamic effects: vibrations in the test system producing oscillations in the recorded quantities, and inertial loads producing forces on the test specimen different from the forces sensed by the test fixture. These effects need either to be controlled and, if possible, reduced by appropriate action, or else to be taken into account through proper analysis of the measured data.

The relative importance of such effects increases with increasing testing rate (decreasing test duration). At speeds of less than 0,1 m/s (loading times of greater than 10 ms) the dynamic effects may be negligible and the testing procedure given in ISO 13586 can be applied as it stands. At speeds approaching 1 m/s (loading times of the order of 1 ms) the dynamic effects may become significant but still controllable. The procedure given in ISO 13586 can still be used though with some provisos and these are contemplated in this document. At speeds of several meters per second and higher (loading times markedly shorter than 1 ms) the dynamic effects become dominant, and different approaches to fracture toughness determination are required, which are outside the scope of this document.

The general principles, methods and rules given in ISO 13586 for fracture testing at low loading rates remain valid except where expressly stated otherwise in this document.

The methods are suitable for use with the same range of materials as covered by ISO 13586, i.e.

— rigid and semi-rigid thermoplastic moulding, extrusion and casting materials;

— rigid and semi-rigid thermosetting moulding and casting materials;

and their compounds containing fibres ≤ 7,5mm in length.

In general, fibres 0,1 mm to 7,5 mm in length are known to cause heterogeneity and anisotropy, especially significant in the fracture processes. Therefore, in parallel with Annex B of ISO 13586:2018, where relevant Annex C of this document offers some guidelines to extend the application of the same testing procedure, with some reservations, to rigid and semi-rigid thermoplastic or thermosetting plastics containing such short fibres.

Although the dynamic effects occurring at high loading rates are largely dependent on the material tested as well as on the test equipment and test geometry used, the guidelines given here are valid in general, irrespective of test equipment, test geometry and material tested.

The same restrictions as to linearity of the load-displacement diagram, specimen size and notch tip sharpness apply as for ISO 13586.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 13586:2018, *Plastics — Determination of fracture toughness* (G_{IC} *and* K_{IC}) *— Linear elastic fracture mechanics (LEFM) approach*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 13586 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Test specimens

4.1 Specimen geometry and preparation

As for the low-rate testing case covered by ISO 13586, two test configurations are recommended, namely the three-point bending [also called single edge notch bend (SENB)] and the compact tension (denoted CT), see Figure 1.

Shape and size, preparation, notching and conditioning of test specimens shall comply with the requirements set out in Clause 4 of ISO 13586:2018.

4.2 Crack length and number of test replicates

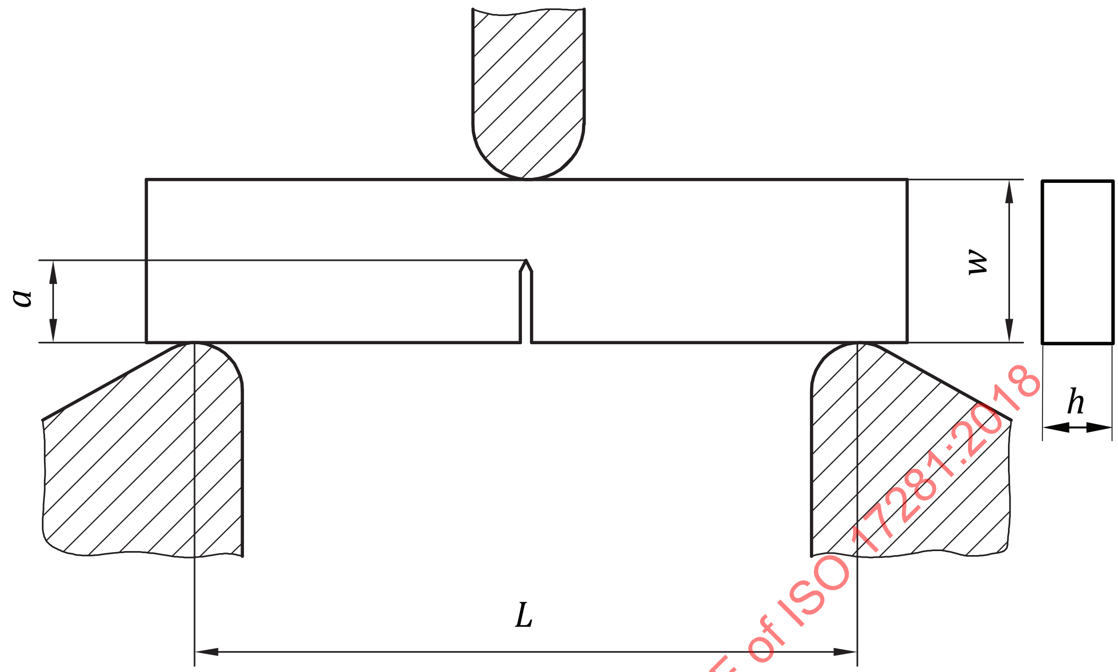
4.2.1 Determination of K_{IC}

As in the low-rate testing case covered by ISO 13586, measuring test specimens having the same crack length is adequate for determining K_{IC}. The initial crack length a should be in the range $0,45 \leq a/w \leq 0,55$. However, in view of the lower degree of accuracy to be expected with measurements at high rates of loading as compared with low-rate testing, it is recommended that at least five replicates, with crack lengths in the range specified above, be used to determine K_{IC}, and the results averaged.

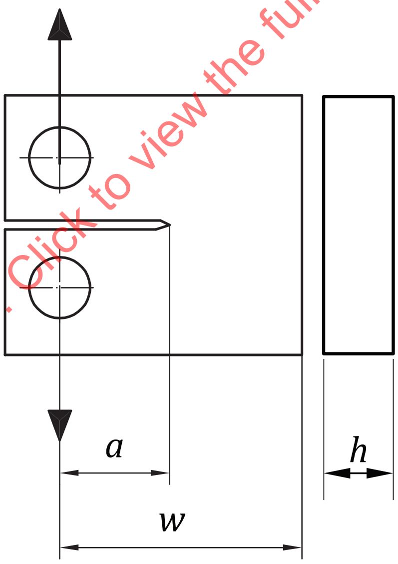
4.2.2 Determination of G_{IC}

At variance with the low-rate testing case covered by ISO 13586, a multi-specimen procedure, using a series of test specimens with identical dimensions but varying crack-length as specified below, shall be applied for determining G_{IC}.

At least 15 valid determinations shall be made, with initial crack length varying over the range $0,20 \leq a/w \leq 0,70$ for the SENB configuration and $0,40 \leq a/w \leq 0,75$ for the CT configuration. They may include the five determinations made on test specimens having initial crack lengths in the range $0,45 \leq a/w \leq 0,55$ to obtain K_{IC}. It is then suggested that, of the remaining ten test specimens to be used, six have initial crack lengths in the range $0,20 \leq a/w \leq 0,45$ and four in the range $0,55 \leq a/w \leq 0,70$ in the case of the SENB configuration and three have initial crack length in the range $0,40 \leq a/w \leq 0,45$ and seven in the range $0,55 \leq a/w \leq 0,70$ in the case of the CT configuration.



a) SENB



b) CT

Key

L span between supports
w specimen width
h specimen thickness
a crack length

Figure 1 — Test configurations as specified in 4.1 and 6.2

4.3 Measurement of test specimen dimensions

Measurement is carried out as described in 5.7 of ISO 13586:2018.

5 Test conditions

5.1 Loading mode

The test shall be performed at constant load-point displacement rate. A maximum variation of 10 % in the load-point displacement rate during the test is allowed (see 6.1).

5.2 Test speed

As a basic test condition, it is recommended that a load-point displacement rate of 1 m/s be used. If a different rate is applied, it shall be quoted in the test report.

With rate-sensitive materials such as plastics, a more significant measure of the rate of the experiment is probably its duration, i.e. the time required to bring the test specimen to fracture. The time to fracture, t_f, is understood here as the time interval between the moment when the load starts acting on the test specimen and the point of fracture initiation as defined in 8.1.

With a fixed load-point displacement rate the time to fracture varies with material and specimen geometry. If results at a given time to fracture (e.g. 1 ms) are desired, it is necessary to adapt the load-point displacement rate of the test to each material and specimen geometry (type and dimensions). For this purpose it is expedient to run some preliminary trial tests at different testing speeds (i.e. load-point displacement rates) to determine the testing speed required to obtain the assigned time to fracture under the given test conditions.

In any case, the time to fracture, t_f, shall also be quoted in the test report.

5.3 Test atmosphere and temperature

These are determined as described in 5.6 and 5.8 of ISO 13586:2018.

6 Test equipment

6.1 Loading machine

Any type of loading machine (impact pendulums, falling-weight towers, servo-hydraulic universal testing machines, etc.) is permitted, provided it is capable of applying an adequate load to bring the test piece to fracture at the required load-point displacement rate and of maintaining this rate constant throughout the test up to fracture initiation. With testing machines of limited capacity, this requirement may need to be verified by preliminary tests, especially when new materials are tested or when new test conditions (e.g. change in specimen size) are used.

Any variation in the load-point displacement rate during the test shall be determined and quoted if it exceeds 10 % of the rate at fracture initiation.

6.2 Loading rigs

Unlike for low-rate testing, the use of fixed anvils rather than moving rollers is preferred for conducting three-point bend (SENB) fracture tests under high rate conditions, as is normally the case with standard impact pendulums. The span between the supports shall be adjustable however, so that specimens of different size can be accommodated, as specified in Clause 4 of ISO 13586:2018.

NOTE In the case of three-point bend testing (SENB specimens), improved results can be obtained if the test piece is held in contact with the anvils by light springs (e.g. rubber bands). These will assist in maintaining the test piece in position during the sudden load transmission from the machine to the test specimen, and ensure more reproducible records.

6.3 Instrumentation

Acquisition of a complete record of the load/time response of the material sample under test is essential for the determination of K_{IC}. In addition, a means of evaluating the displacement of the moving load-point during the test is necessary for the independent determination of G_{IC}. Instrumentation of the testing machine should thus comprise, basically, a force sensing and recording system and a displacement measuring and recording system or devices to measure and record quantities from which the load and the load-point displacement can also be indirectly determined.

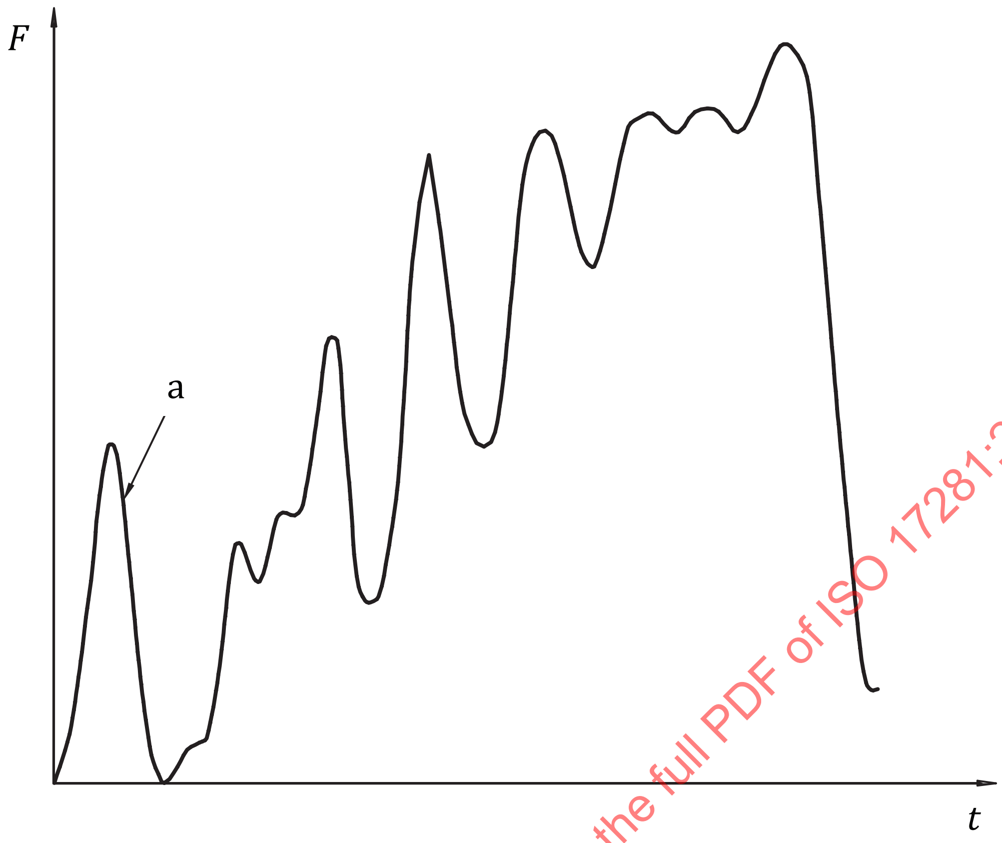
The adequacy of the response of this equipment to the dynamic events occurring in the relevant determinations shall be checked. It can be considered satisfactory if a plain plastic specimen (without any mechanical damping device in place) shows an inertial peak (see Figure 2) larger than 100 N at 1 m/s test speed. The response time shall be <20 % of the input signal rise time.

If a digital recording system is used, the sampling time should be less than 1/200 of the time to fracture, i.e. at least 200 data points should be collected over the time interval from the first increase of the signal to the point of fracture initiation in order to define the required data curve with sufficient accuracy.

7 Control of dynamic effects

7.1 Electronic filtering

The first manifestation of dynamic effects is the presence of oscillations in the load-recording signal. They may complicate the interpretation of the test records up to the point of obscuring the basic response of the specimen under test. It is thus desirable that these effects be contained. Reducing these oscillations artificially, *a posteriori*, by electronic filtering or attenuation can be fallacious however, since it may wipe out some real features of the specimen response. Therefore, electronic filtering or attenuation is not permitted unless the source of the removed "noise" is known and the effect on the data is understood.



Key

F load

t time

a Inertial peak.

Figure 2 — Typical load/time record in the absence of signal attenuation and mechanical damping

7.2 Mechanical damping

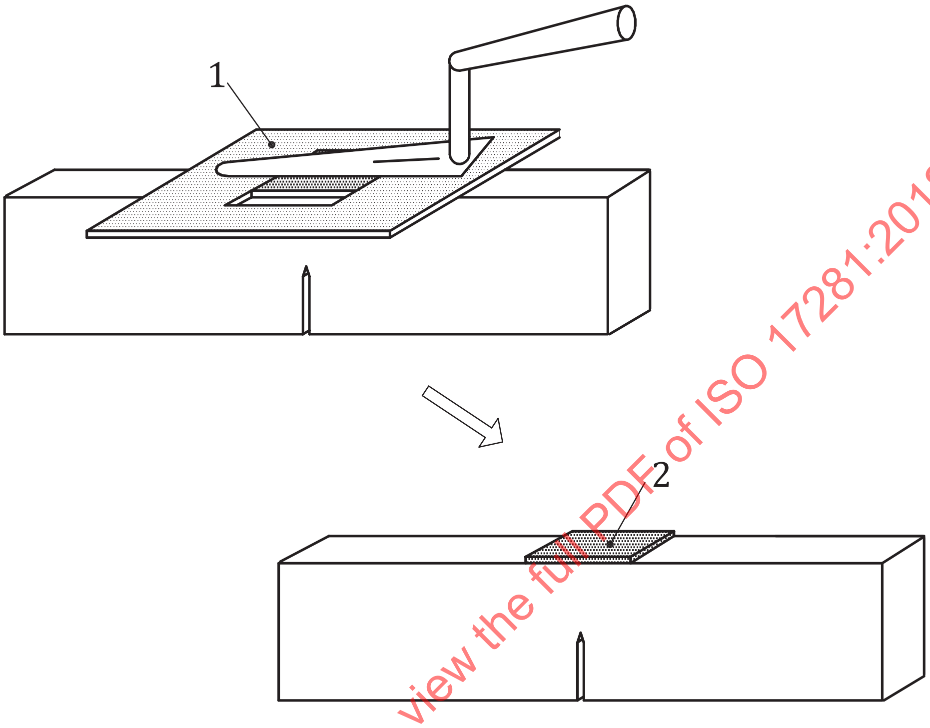
Some control of the effects of inertial loads can be achieved by proper mechanical damping of the load transmission. With impact testing machines the impact may be cushioned by means of a soft pad, placed where the tup strikes the specimen. The pad should reduce the inertial effects by reducing the “contact stiffness”. With high-speed testing machines (e.g. servo-hydraulic), initial acceleration of the specimen can be controlled by means of a damper applied in the motion transmission unit.

With impact testing machines and SENB test specimens the damping pad can be made by spreading a layer of a paste or a highly viscous grease over the contact surface either of the tup of the striking hammer or of the test piece. For the sake of reproducibility it is important that the grease be homogeneous and evenly applied, with thickness constant to ±0,05 mm. This can be obtained by delivering the grease with a spatula through an aluminium stencil having the required thickness, normally a few tenths of a millimetre, as shown in Figure 3.

With high-speed testing machines and CT test specimens the damping pad can be more conveniently made of a viscoelastic rubber-like material with a low coefficient of restitution. The rubber-like character should ensure a more or less complete recovery of the pad deformation after each test, thus allowing the same pad to be used repeatedly.

7.3 Damping level

If mechanical damping is applied, it shall be kept to a minimum, sufficient to contain the fluctuations in the force-time trace within the 10 % envelope defined in 8.1. To obtain this optimal result it is advisable to run some preliminary trial tests to gauge the performance of the damper. This can be varied by changing the consistency of the damping material used and/or the thickness of the pad made thereof.



Key

1 aluminium stencil

2 damping pad

Figure 3 — Deposition of damping pad on SENB test specimen

If the test specimens are in short supply, it is advisable to use an unnotched specimen to assay the performance of the damper. The dynamic effects that are to be controlled by mechanical damping are in fact largely independent of crack length and the use of an unnotched specimen offers the advantage that it can stand repeated strokes without breaking.

In order to determine the level of damping needed to meet the requirement stated in 8.1, reference should be made to the worst case to be expected in the testing programme, i.e. the case of the specimen with the deepest notch, which will present the lowest fracture resistance and thus the largest (force oscillation)/(fracture load) ratio.

7.4 Check on speed

Because of damping, some deviations from the pre-set load-point displacement rate may ensue. Thus, if mechanical damping is applied, the instrument shall be reset to the desired load-point displacement rate and its constancy checked (as requested under 5.2) under the actual test conditions, i.e. with the damping device in place.

If mechanical damping is applied, it shall be recorded in the test report.

8 Data handling

8.1 Analysis of the test records and identification of fracture initiation

These tests, as well as the low speed tests covered in ISO 13586, are designed to characterize the toughness at fracture initiation. Once a fracture test has been performed and the load-time or load-load point displacement curve has been obtained, the question arises of identifying the point of fracture initiation. Several techniques are possible, but in this document it is deduced from the load diagram.

The same rules as those stated in ISO 13586, for the determination of F_Q are used here, but in the case of high-rate testing some preliminary analysis of the load-time record is required to make sure that dynamic effects do not obscure the basic response of the specimen under test.

Firstly, in the case of high-rate testing, a load drop before maximum load should not be assumed to be an arrested crack extension ("pop-in"), unless borne out by examination of the fracture surface.

Secondly, the occurrence of force peaks and fluctuations in the initial part of the load-time record is tolerated, but a limit is placed on force fluctuations in the portion of the force-time record where the force exceeds 1/2 of its value at fracture initiation and the curve is smoothed. The linearity requirements referred to in 6.1 of ISO 13586:2018 need to be verified here on the "smoothed" load-displacement curve.

The procedure is as follows.

Draw a smooth mean force-time curve through the experimental load-time record, $F(t)$, and determine F_{max} and F_{max} /3 on that curve (see Figure 4). Then improve the determination of the mean load/time curve by a computer-aided curve-fitting procedure. The following empirical fitting formula is suggested:

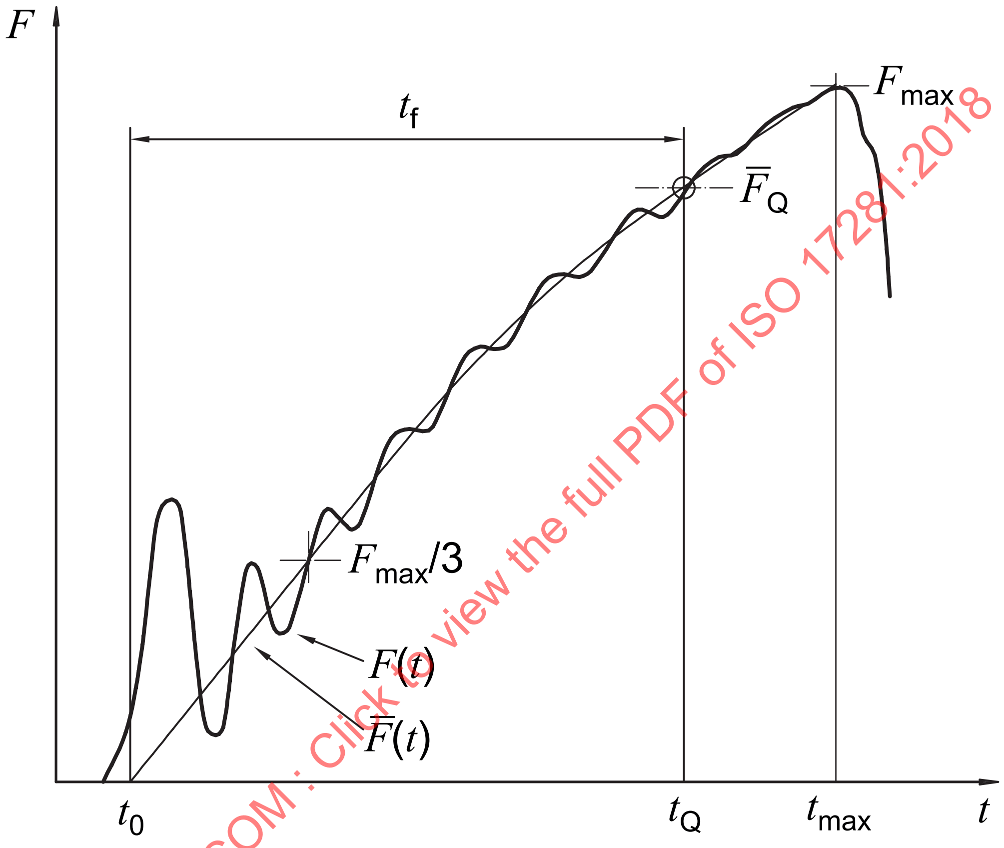
$$\bar{F}(t) = m(t - t_0) - b(t - t_0)^n \quad (1)$$

where t_0, m, b and n are (positive) fitting parameters, with n preferably ≥5.

Use the curve drawn previously to obtain a first estimate of these parameters (see Annex A) and use this set of values at the start of the regression analysis. The regression analysis should be confined to the portion of the experimental curve comprised in the time interval defined by F_{max} / 3 and F_{max}. The value of the initial time, t_0, should also be derived from the regression analysis. However, if that value turns out to be smaller than the time when the force signal first rises, take the latter one as initial time t_0 and repeat the curve fitting by forcing the new curve $\bar{F}(t)$ to pass through the point $t = t_0$, $F = 0$. Finally, determine $\bar{F}_Q$ on the curve $\bar{F}(t)$ (Figure 4), as indicated in 6.1 of ISO 13586:2018 (see also the Note below). To this end, the "maximum load" — to be denoted $\bar{F}_{max}$ — is defined here as the value of the fitted force, $\bar{F}(t)$, at time $t = t_{max}$ corresponding to the maximum of the experimental curve (see Figure 4).

The curve $\bar{F}(t)$ so obtained is assumed to be a good representation of what the load-time response of the system would be in the absence of dynamic effects, provided it meets the following requirement (see Figure 5): the force $F(t)$ recorded experimentally shall not deviate from the mean current value $\bar{F}(t)$ by more than 5 % of the critical value $\bar{F}_Q$ over the time interval defined by $\bar{F}_Q/2$ and $\bar{F}_Q$. To check this draw two lines parallel to the curve $\bar{F}(t)$ at a distance of 5 % of $\bar{F}_Q$ on either side of it, over the time interval defined by $\bar{F}_Q/2$ and $\bar{F}_Q$. All parts of the experimental curve $F(t)$ in that interval should fall within this 10 % envelope. If the experimental curve $F(t)$ fails this requirement, then the determination shall be deemed invalid. Before abandoning any determination however, action shall be taken to try and reduce the dynamic effects further, as stated in Clause 7.

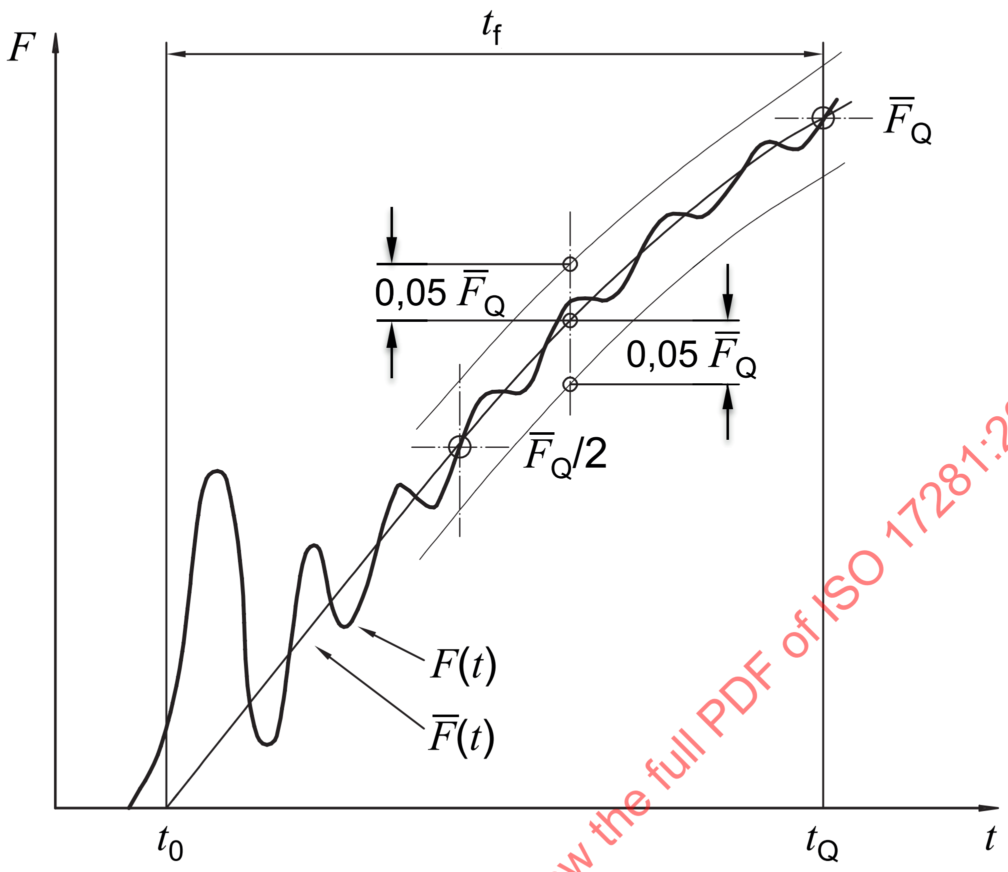
NOTE Once the parameters of the best fit have been determined, the two straight lines to be used in order to identify $\overline{F}_Q$ (see ISO 13586:2018, 6.1) can be simply obtained as given by the formulae $\overline{F} = m(t - t_0)$ and $\overline{F} = 0{,}95m(t - t_0)$ and the value of $\overline{F}_5$ can be readily calculated as $\overline{F}_5 = 0{,}95\, m(0{,}05\, m / b)^{1/(n-1)}$. Furthermore, if $\overline{F}_Q = \overline{F}_5$ then the time to fracture can be calculated as $t_f = t_5 - t_0 = (0{,}05\, m/b)^{1/(n-1)}$.



Key

F	load
t	time
$F(t)$	load-time curve recorded experimentally
$\overline{F}(t)$	load-time fitted curve
F_{max}	maximum load
t_{max}	time at maximum load
$\overline{F}_Q$	load at fracture initiation;
t_Q	time at $F = \overline{F}_Q$
t_0	initial time
t_f	time to fracture

Figure 4 — Schematic representation of curve fitting and determination of $\overline{F}_Q$ and t_f



Key

F load

t time

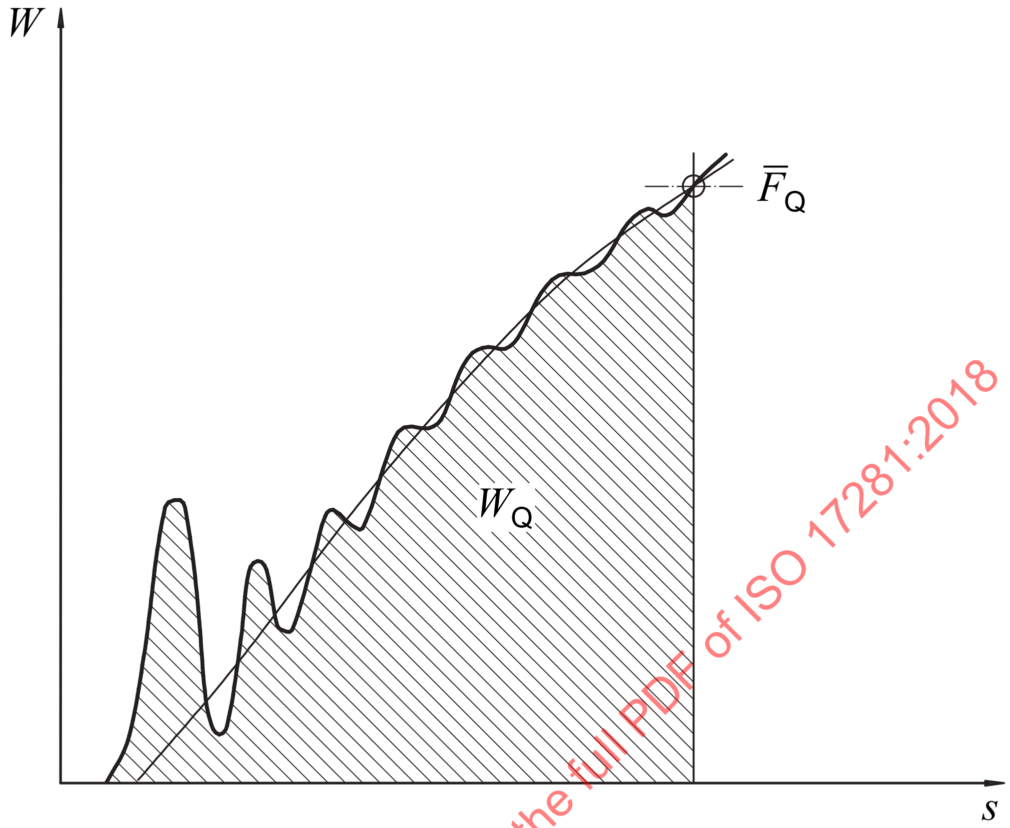
Other symbols are as per Figure 4

Figure 5 — Schematic representation of limits of permissible force fluctuations in the fracture test

8.2 Energy correction

8.2.1 General

As in the low-rate testing case covered in ISO 13586, G_{IC} shall be determined directly from the energy derived from integrating the load-load point displacement diagram. As in the low-rate case however, the area W_Q under the measured load-load point displacement curve (Figure 6) contains extraneous contributions in excess of the true fracture energy, W_B, and some corrections are required before G_{IC} can be calculated from that energy. As a matter of fact, unless an external displacement measuring device is used (e.g. optical), the apparent load point displacements are in excess of the specimen deformation. Besides indentation of the test piece and compliance of the testing machine, the compression of the mechanical damping device (if used) also contributes to this excess. Correction for these effects is covered in 8.2.2. Moreover, in the case of high-rate testing, the area W_Q under the measured load-load point displacement curve also contains some contributions from the kinetic energy (U_{kin}) of the moving test specimen and from inertial loads (U_{inert}) produced by test piece acceleration. A procedure to get rid of these parasitic energy terms is described in 8.2.3.



Key

F load

s load point displacement

$\overline{F}_Q$ load at fracture initiation

W_Q overall energy input up to fracture initiation

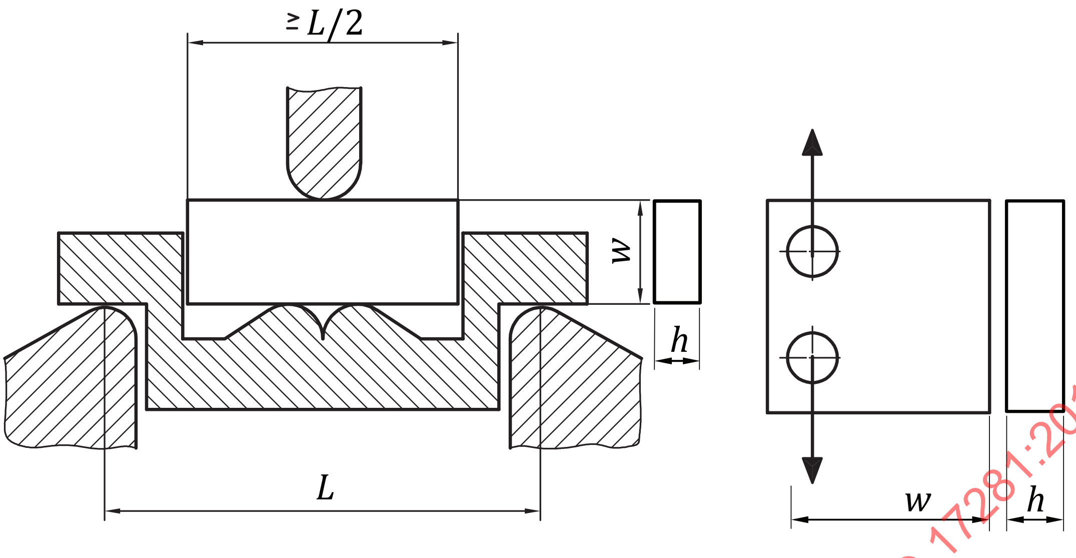
Figure 6 — Evaluation of energy W_Q from the fracture test

8.2.2 Test piece indentation, machine compliance and damper compression

The correction for test piece indentation, machine compliance and damper compression can be estimated from a separate test, to be performed on an unnotched specimen, as specified in 5.5 of ISO 13586:2018. Suggested unnotched specimen arrangements for the correction test are shown in Figures 7 a) and b), for SENB and CT configurations, respectively.

It is advisable to carry out two or three replicates of the correction test in order to check repeatability and, in case of large variations, to check for possible errors.

The force/displacement correlation obtained in the correction test is integrated up to the initiation load $\overline{F}_Q$ determined in the fracture test (see Figure 8) and the obtained energy W_{cor} is subtracted from the energy W_Q obtained by integrating the force/displacement curve measured in the fracture test (see Figure 6).



a) **SENB configuration** b) **CT configuration**

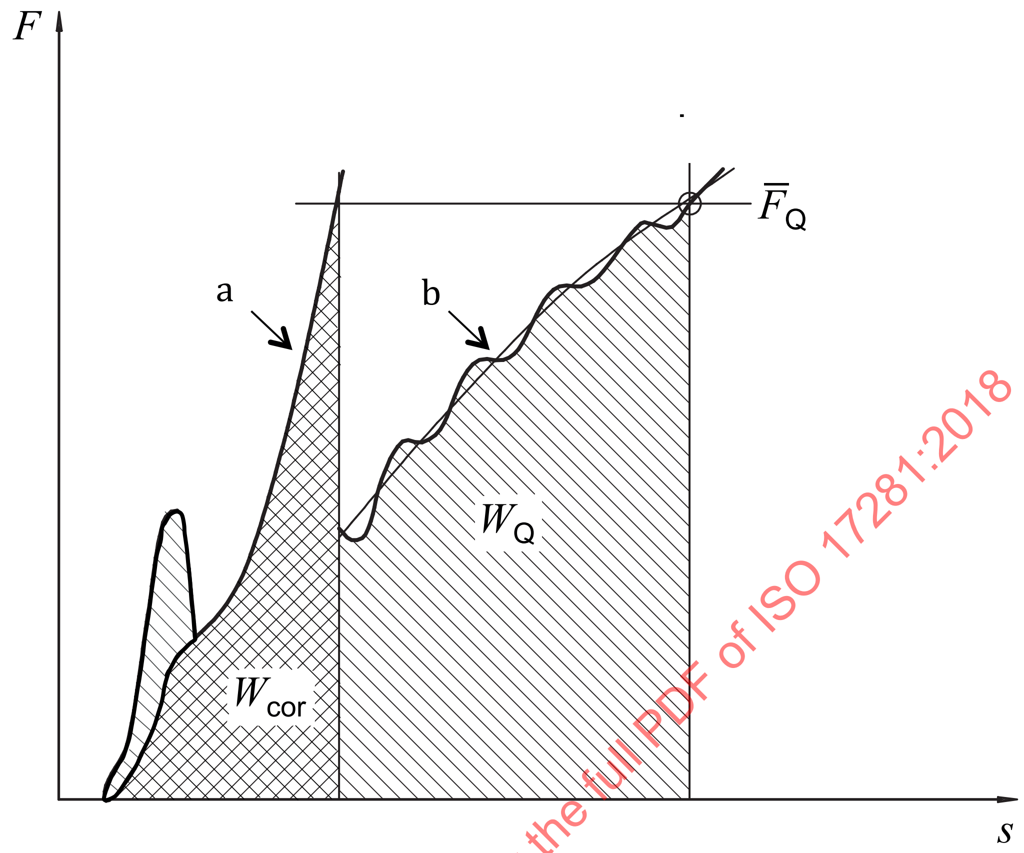
Key

L span between supports in the fracture test

w specimen width

h specimen thickness

Figure 7 — Arrangements for the energy correction test



Key

F	load	W_Q	overall input energy up to fracture initiation
$\bar{F}_Q$	load at fracture initiation	W_{cor}	energy correction
s	load point displacement	a	Correction test.
		b	Fracture test.

Figure 8 — Evaluation of energy W_{cor} from the correction test as specified in 8.2.1 (the plots obtained from the correction test and the fracture test are shown superposed)

The magnitude of this correction, W_{cor}, depends on the magnitude of $\bar{F}_Q$, which may vary substantially from specimen to specimen, especially if the initial crack length to width ratio, a/w, varies. The correction should therefore be computed for each specimen subjected to the fracture test, and applied to its respective total energy to fracture, W_Q.

As specified in 5.5 of ISO 13586:2018, the correction test shall be performed such that the loading time (up to load $\bar{F}_Q$) is the same as in the fracture test, i.e. t_f. This will involve lower test speeds to reach the same load in the same time, e.g. about half the speed of the fracture test. Furthermore, with specimens of varying crack length this requirement would imply performing different correction tests at different speeds. This is deemed unnecessary provided time-to-fracture variations among the given set of specimens are less than 50 % of the mean time-to-fracture; it is then sufficient to perform the correction test at a mean testing speed.

Because of the damper compression contribution the correction may be substantially larger than it is in the low-rate tests, where no damping is applied. Moreover, because of dynamic effects and the effect of mechanical damping the load-load point displacement curve obtained in the correction experiment is seldom linear, and the practice of linearizing the near-zero data before evaluating displacement or compliance corrections, as suggested in ISO 13586, is not advisable. It is preferable to follow the alternative way of correcting energies, as stated above.

8.2.3 Kinetic energy and inertial loads

The corrected energy $W_{Q,cor} = (W_Q - W_{cor})$ should be further diminished by the two aforementioned parasitic energy terms, U_{kin} and U_{inert}, to obtain the true fracture energy W_B from which G_{IC} can be determined.

An alternative route that completely circumvents the need to evaluate that correction consists of determining G_{IC} from the slope of a plot of fracture energy versus the energy calibration factor ϕ (or the product $hw\phi$) [see Formula (6) of ISO 13586:2018 and Figure 9 a) of the present document] obtained by testing a series of specimens with equal dimensions but varying crack length. Since U_{kin} and U_{inert} are essentially independent of crack length, their addition to the fracture energy W_B in an energy versus ϕ (or versus $hw\phi$) graph will not alter the slope, and no correction is necessary [see Figure 9 b)].

NOTE Subtraction of W_{cor} from W_Q does not get rid of the kinetic and inertial contributions contained in W_Q. As a matter of fact, when W_{cor} is measured (correction test) specimen's motion is essentially suppressed and inertial effects are substantially reduced compared with the inertial effects occurring in the fracture test, as a result of the reduced speed used in the correction test (see 8.2.1).

9 Expression of results

9.1 Determination of K_{IC}

The value of $\overline{F}_Q$ determined as specified in 8.1 is used to calculate K_Q as specified in 6.3 of ISO 13586:2018.

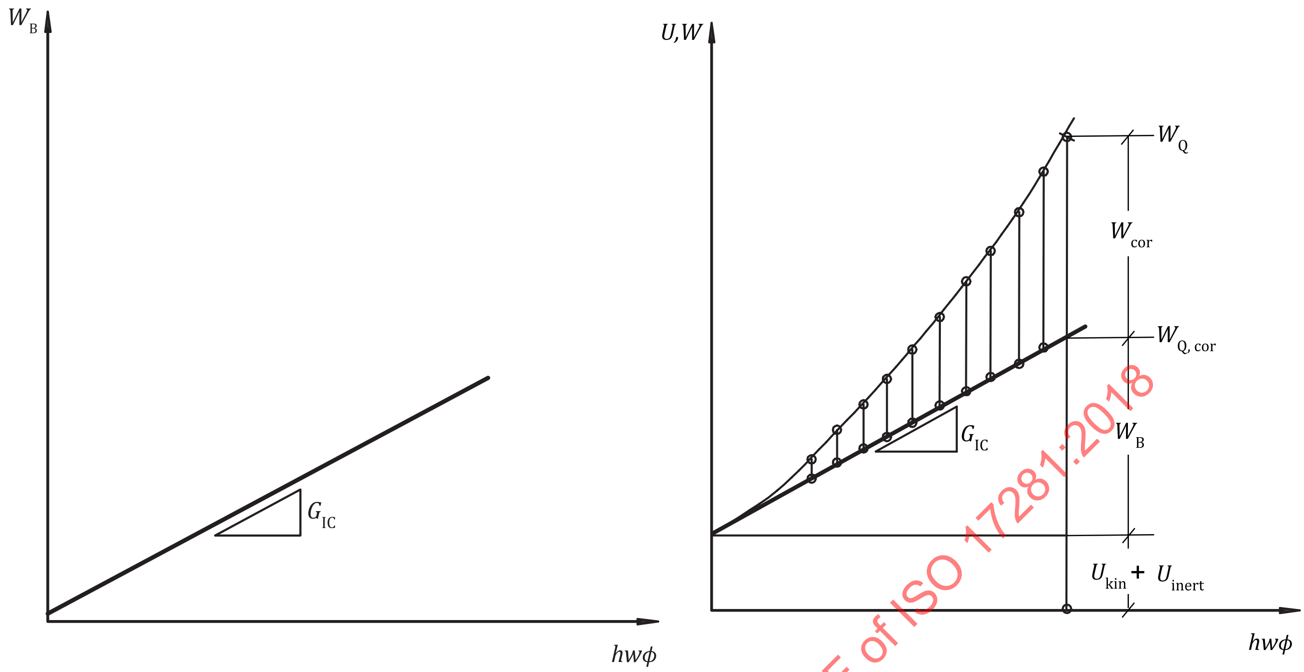
The provisional value, K_Q, shall be checked for linearity and size requirements according to the criteria stated in 6.4 of ISO 13586:2018 before it can be assumed to be a valid K_{IC} value. For the linearity criterion, the "maximum load" that $\overline{F}_Q$ is to be confronted with is the value $\overline{F}_{max}$ defined in 8.1.

The time to fracture, t_f, is then evaluated as the difference $t_f = t_Q - t_0$ between the time at the instant when the load is $\overline{F}_Q$ and the initial time t_0 as determined above.

9.2 Determination of σ_y

The uniaxial tensile yield stress, σ_y, to be used in the size validity criteria should be determined under loading rate conditions comparable to those in the fracture test: the tensile test can be performed at a constant stroke-rate such that the loading time to yield, t_y, is within ±20 % of the actual loading time observed in the fracture test, t_f.

Since σ_y is a decreasing function of time, a low-rate value may be used in the first instance to give a conservative size value. If the result is valid, it is then unnecessary to measure σ_y under high-rate conditions. If the result is invalid, determine and use the high-rate σ_y value.



a) From fracture energy, W_B plotted versus $hw\phi$

b) From corrected energy, $W_{Q,cor} = (W_Q - W_{cor})$ plotted versus $hw\phi$

Key

W_B fracture energy

U, W various contributions to total energy W_Q (see text)

$hw\phi$ normalized specimen cross-section area (see text)

G_{IC} fracture toughness

Figure 9 — Determination of G_{IC}

If a high-rate testing machine is not available for the tensile test, σ_y may be determined by extrapolation of values obtained from low-rate tests covering a range of times to yield, on a logarithmic time-scale.

The method of finding σ_y shall be quoted in the test report.

9.3 Determination of G_{IC}

Produce a series of test specimens with equal dimensions but varying crack length and test them under equal conditions (including damper characteristics and testing rate). Determine $\overline{F}_Q$ for each individual test specimen as specified in 8.1 and check for its validity (linearity and size criteria) as specified in 9.1.

Determine W_Q for each individual test specimen by integrating the respective load-load point displacement diagram up to the load point ($\overline{F}_Q$) defining fracture initiation (see Figure 6).

Determine the energy correction, W_{cor} for each individual test specimen by integrating the load-load point displacement diagram of the correction test up to $\overline{F}_Q$ (see Figure 8).

Plot the corrected energies, $W_{Q,cor} = (W_Q - W_{cor})$, as a function of $hw\phi$ and best fit a straight line through the data points [see Figure 9 (b)]. From the slope of this line the value of G_{IC} is determined.

The parasitic energy contribution ($U_{kin} + U_{inert}$) mentioned above will appear on this plot as a positive intercept of the regression line on the energy axis. If a negative intercept is obtained then the results should be examined for possible errors.

If results at a fixed time to fracture are desired, specimens of varying crack length should be tested under different testing speeds (i.e. load point displacement rates) in order to obtain the same time to fracture. If the same test speed is used and the effect of varying time to fracture is neglected, the resulting G_{IC} value should be quoted in association with the mean time to fracture obtained in the K_{IC} determination.

In view of the difficulty in determining the correct specimen compliance under high-rate conditions, the cross check on accuracy via $E_t/(1 - \mu^2)$ suggested in 6.5 of ISO 13586:2018, should not be applied here. The value of $K_{IC}{}^2/G_{IC}$ should still be reported for the sake of information.

10 Precision

Tables 1 and 2 give sets of data obtained on two representative materials producing a loading curve (mean line) nearly linear up to the maximum force and a loading curve (mean line) slightly curved, respectively.

Table 1 gives the set of data obtained by twelve laboratories on a polyvinylchloride (PVC) sample. All the data were obtained in SENB testing and three types of testing machine were used: impact pendulum, falling weight and servohydraulic testing instrument. Fracture initiation was generally identified by the point of maximum force (column four). The means of the K_{IC} values obtained from valid tests at $a/w \sim 0{,}5$ are given together with the partial standard deviations (column eight). The slope of the linear regression through the corrected energy values obtained from valid tests covering a range of a/w is given for G_{IC} (column eleven). The standard deviation from the mean values of all participating laboratories (bottom lines) is 5 % for K_{IC} and 18 % for G_{IC}.

Table 1 — K_{IC} and G_{IC} measurements on PVC

Lab. No.	Testing machine	Specimen type	F_5 or F_{max}	Mean t_f ms	K_{IC} determination			G_{IC} determination			$K_{IC}{}^2/G_{IC}$ GPa
					Valid tests	a/w	K_{IC} MPa·m$^{1/2}$	Valid tests	a/w range	G_{IC} kJ/m^2	
1	Falling weight	SENB	max.	0,73	5	0,54	2,70 ± 0,26	9	0,20 to 0,71	1,47	4,96
2	Servohydraulic	SENB	max.	0,78	5	0,50	2,65 ± 0,10	15	0,20 to 0,70	2,19	3,21
3	Pendulum	SENB	max.	0,60	6	0,50	2,60 ± 0,17	13	0,19 to 0,69	1,63	4,15
4	Pendulum	SENB	max.	0,56	3	0,49	2,45 ± 0,07	12	0,20 to 0,70	1,50	4,00
5	Servohydraulic	SENB	max.	0,70	5	0,50	2,76 ± 0,13	15	0,20 to 0,71	2,00	3,81
6	Falling weight	SENB	max.	0,63	5	0,51	2,53 ± 0,02	15	0,20 to 0,70	1,90	3,37
7	Falling weight	SENB	max.	0,92	5	0,51	2,51 ± 0,13	14	0,20 to 0,65	1,45	4,34
8	Falling weight	SENB	5 %	0,63	4	0,50	2,82 ± 0,27	13	0,25 to 0,70	1,85	4,30
9	Falling weight	SENB	max.	0,82	3	0,50	2,59 ± 0,08	11	0,20 to 0,65	1,25	5,37
10	Servohydraulic	SENB	max.	0,81	4	0,50	2,56 ± 0,12	11	0,20 to 0,72	(3,23)[a]	—
11	Servohydraulic	SENB	max.	1,27	5	0,52	2,82 ± 0,28	15	0,21 to 0,71	(5,08)[b]	—
12	Servohydraulic	SENB	max.	0,50	1	0,52	2,88	8	0,21 to 0,62	(5,00)[b]	—
					mean		2,66 ± 0,15	mean		1,69	
					standard deviation		0,14 (5 %)	standard deviation		0,31 (18 %)	

[a] Error suspected.

[b] Without energy correction.

Table 2 gives the set of data obtained by ten laboratories on a rubber-modified poly(methyl methacrylate) (PMMA-RT) sample. Most data were obtained in SENB testing and three types of testing machine were used: impact pendulum, falling weight and servo-hydraulic testing instrument. Fracture initiation was mostly identified with the 5 % offset (column four). The means of the K_{IC} values obtained from valid tests at $a/w \sim 0{,}5$ are given together with the partial standard deviations (column eight). The slope of the linear regression through the corrected energy values obtained from valid tests covering a range of

a/w is given for G_{IC} (column eleven). The standard deviation from the mean values of all participating laboratories (bottom lines) is 8 % for both K_{IC} and G_{IC}.

Table 2 — K_{IC} and G_{IC} measurements on PMMA-RT

Lab. No.	Testing machine	Specimen type	F_5 or F_{max}	Mean t_f ms	K_{IC} determination			G_{IC} determination			K_{IC}^2/G_{IC} GPa
					Valid tests	a/w	K_{IC} MPa·m$^{1/2}$	Valid tests	a / w range	G_{IC} kJ/m^2	
1	Falling weight	SENB	max. 5 %	0,98	5	0,45	4,16 ± 0,46	7	0,36 to 0,70	4,25	3,58
2	Servohydraulic	SENB	5 %	1,21	5	0,47	3,70 ± 0,04	10	0,16 to 0,65	5,03	2,73
3	Pendulum	SENB	5 %	1,12	5	0,49	3,42 ± 0,09	14	0,25 to 0,69	4,08	2,87
4	Servohydraulic	SENB	5 %	1,20	5	0,51	3,71 ± 0,08	11	0,36 to 0,66	4,23	3,19
5	Falling weight	SENB	5 %	0,96	5	0,50	4,12 ± 0,05	11	0,35 to 0,65	4,20	3,90
	Falling weight	SENB	5 %	1,00	5	0,50	4,13 ± 0,07	11	0,35 to 0,65	4,70	3,43
	Falling weight	SENB	5 %	1,00	5	0,50	3,92 ± 0,02	12	0,30 to 0,65	4,30	3,48
6	Servohydraulic	SENB	5 %	1,10	5	0,50	4,20 ± 0,45	10	0,36 to 0,60	4,67	3,78
7	Servohydraulic	SENB	max. 5 %	1,03	9	0,49	4,21 ± 0,16	23	0,19 to 0,69	4,95	3,58
8	Servohydraulic	SENB	max.	1,32	5	0,49	4,49 ± 0,04	15	0,17 to 0,70	4,71	4,21
9	Servohydraulic	SENB	max.	0,66	3	0,50	3,71 ± 0,23	5	0,28 to 0,50	(5,54)[a]	—
10	Servohydraulic	CT	5 %	1,06	5	0,51	3,71 ± 0,06	15	0,31 to 0,71	(8,44)[b]	—
					mean		3,96 ± 0,15	mean		4,51	
					standard deviation		0,31 (8 %)	standard deviation		0,34 (8 %)	

[a] Without energy correction.

[b] Error suspected.

11 Test report

The test report shall contain the following information:

a) reference to this document, i.e. ISO 17281:2018;

b) all details necessary for complete identification of the material tested, including source and history;

c) test specimen shape (SENB or CT) and dimensions;

d) notching method used;

e) test temperature and speed;

f) type of test apparatus used;

g) type of mechanical damping device used (if any);

h) maximum test speed variation during the tests (if in excess of 10 %);

i) an example of load-time or load-displacement curve, showing the 10 % envelope and $\bar{F}_Q$ determination;

j) number of specimens tested and the ranges of crack length used for determining K_{IC} and G_{IC} respectively;

k) kind of initiation point (pop-in, 5 % offset or maximum load) and the ratio $\bar{F}_{max} / \bar{F}_5$, if relevant;

l) time to fracture;

m) yield stress determination procedure used and the loading time;

n) results of the size criteria assessment;

o) diagram of energies W_Q and $W_{Q\ cor}$ versus $hw\phi$;

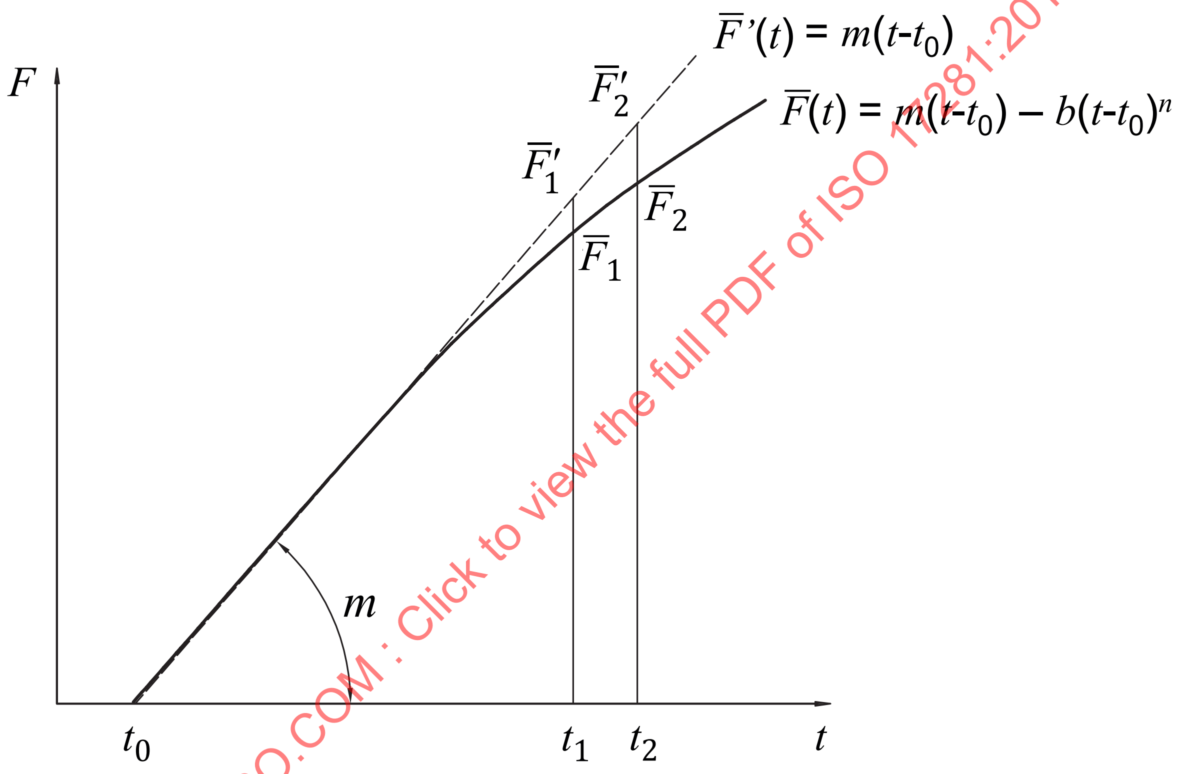
p) critical stress intensity factor K_{IC} and critical energy release rate G_{IC};

q) value of K_{IC}^2/G_{IC}.

Annex A
(informative)

Estimation of curve fit parameters

Once a smooth mean force-time curve $\bar{F}(t)$ has been drawn by guesswork (full line in Figure A.1), the values of the two parameters characterizing the initial, linear portion of the curve, i.e. initial time t_0 and initial slope *m*, can be evaluated from the initial tangent $\bar{F}'(t)$ (dashed line in Figure A.1).



Key

F	load
t	time
$\bar{F}(t)$	fitting load-time curve
$\bar{F}'(t)$	initial tangent
m	initial slope
m, t_0, *b*, *n*	fitting parameters

Figure A.1 — Construction for the estimation of the curve fitting parameters t_0, *m*, *b*, *n*

The values of the two parameters, *b* and *n*, which characterize the deviation from linearity can then be estimated as follows.

Draw two vertical lines through the curved portion of the $\bar{F}(t)$ curve (e.g. at times t_1 and t_2) and measure the two segments $\bar{F}_1\bar{F}_1'$ and $\bar{F}_2\bar{F}_2'$ (see Figure A.1), then n is calculated from

$$n = \frac{\ln[(\bar{F}_2' - \bar{F}_2)/(\bar{F}_1' - \bar{F}_1)]}{\ln[\bar{F}_2'/\bar{F}_1']} \quad \text{(A.1)}$$

and b is obtained from

$$b = m(t_1 - t_0)^{1-n} - \bar{F}_1(t_1 - t_0)^{-n} \quad \text{(A.2)}$$

Annex B
(informative)

Recommended test report forms

Recommended test report form ISO 17281, page 1 of 5 Form (a)

Name:	**Date of testing:**
Organization:	**ISO Standard:**

Material:	Temperature [°C]:

GENERAL TEST CONDITIONS

Test equipment characteristics

1.1. Type of testing apparatus:

1.2. Test fixture (if different from that stated in the standard):

1.3. Instrumentation:

1.4. Quantities monitored:

1.5. Sampling time t_s [µs]:

Test performance

2.1. Inertial peak height (without damping): >100 N?

2.2. Mechanical damping device used (if any):

2.3. Load-point displacement rate variation during the test: <10 %?

2.4. Minimum time to fracture recorded, $t_{f\,min}$ [ms]:

2.5. Minimum number of data points between t_0 and t_Q (i.e. $t_{f\,min}/t_s$): >200?

Data handling

3.1. Determination of $\overline{F}_Q$: curve regression analysis applied successfully?

3.2. Yield stress determination procedure used:

Remarks (Any deviation from procedure and conditions stated in the International Standard):

Recommended test report form ISO 17281, page 2 of 5 Form (b)

Name:	**Date of testing:**
Organization:	**ISO Standard:**

Material:	Temperature [°C]:

K_{IC} DETERMINATION

	Notching method:		root radius: [μm]							
1		Specimen N°							Mean	St.dev.
2	Specimen type (SENB or CT?)	dimensions	h [mm]							
3			w [mm]							
4			a [mm]							
5			a/w [–]							
6	Damping applied?		(Y/N)							
7	Speed		[m/s]							
8	Speed variation: <10 %?		(Y/N)							
9	Load	Fluctuations: within limits?	(Y/N)							
10		$\overline{F}_{max}$	[N]							
11		$\overline{F}_{Q}$	[N]							
12		Q = <pop-in>, <5 %> or <max>?								
13		If Q = <5 %>: $\overline{F}_{max} / \overline{F}_{5}$								
14		<1,1?	(Y/N)							
15	Time to fracture, t_f		[ms]							
16	Geometry calibration factor $f(a / w)$		[–]							
17	$K_Q = f \overline{F}_Q / hw^{1/2}$		[MPa m$^{1/2}$]							
18	Yield	measured or calculated?	(M/C)							
19		σ_y	[MPa]							
20		Time to yield, t_y	[ms]							
21	Size	2,5 $(K_Q/\sigma_y)^2$	[mm]							
22		<h?	(Y/N)							
23		<a?	(Y/N)							
24		<$(w - a)$?	(Y/N)							
25	K_{IC}		[MPa m$^{1/2}$]							
26	Time to fracture, t_f		[ms]							
27	$\dot{K}_{IC} = K_{IC} / t_f$		[GPa m$^{1/2}$s^{-1}]							
28	Energy	W_Q, uncorrected	[mJ]							
29		Energy correction, W_{cor}	[mJ]							
30		$W_{Q,cor} = W_Q - W_{cor}$	[mJ]							
31		Energy calibration factor, $\phi\ (a/w)$	[–]							
32		$hw\phi$	[mm^2]							
33	G_{IC} (from $W_{Q,cor}$ slope)		[kJ m^{-2}]							
34	K_{IC}^2/G_{IC}		[GPa]							

NOTE White cells = to be filled in with experimental data; light grey cells = to be calculated. Heavy grey cells will remain empty.